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Indian Standard SPECIFICATION FOR DRIERS FOR PAINTS

UDC 667-629-3



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INDIAN STANDARDS INSTITUTION
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NEW DELHI 110002

Indian Standard SPECIFICATION FOR DRIERS FOR PAINTS

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(Continued on page 2)

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(Continued from page 1)

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Indian Standard SPECIFICATION FOR DRIERS FOR PAINTS

0. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 25 February 1978, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.
- **0.2** Driers are substances which when incorporated in relatively small proportions in drying oils, or in paints or varnishes based on drying oils, bring about an appreciable reduction in their drying times.
- 0.3 While reviewing IS: 385-1962* and IS: 386-1962† for revision, it was found desirable to amalgamate these two standards. During subsequent discussions it was felt that a broad based specification to cover important metallic driers individually with specific metal content be formulated and accordingly this standard has been prepared. The corresponding methods of test have also been included. An amalgamated revision of IS: 385-1962* and IS: 386-1962† covering mixed driers is under preparation.
- 0.4 This standard contains clauses 4.1.2, 5.1, 5.3 and item (i) of Table 1 which call for agreement between the purchaser and the supplier.
- 0.5 In the preparation of this standard substantial assistance has been derived from the data supplied by National Test House, Calcutta; M/s Technico Enterprises (Pvt) Ltd, Calcutta and M/s Alkali and Chemical Corporation of India Ltd, Calcutta which is gratefully acknowledged.
- **0.6** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS: 2-1960‡. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for octoate and naphthenate driers of cobalt, lead, manganese, zinc, calcium, iron, cerium, barium and zirconium, for paints.

^{*}Specification for liquid driers for paints (revised).

[†]Specification for liquid driers, concentrated, for paints (revised).

[‡]Rules for rounding off numerical values (revised).

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in 2 of IS: 101-1964* and IS: 1303-1963† shall apply.

3. TYPES AND GRADES

- 3.1 The material shall be of two types based on its physical form, namely:
 - a) Type 1 Solid, and
 - b) Type 2 Liquid.
- 3.1.1 The liquid driers shall have three grades, namely, Grade 1, Grade 2 and Grade 3 depending on their metal content.

4. REQUIREMENTS

4.1 Description

- 4.1.1 Solid Driers The material shall be homogeneous, solid or pasty mass and shall be free from dust, grit and other visible impurities.
- 4.1.2 Liquid Driers The material shall be in the form of a solution in petroleum hydrocarbon solvents (conforming to IS: 1745-1966‡) or in some other suitable organic solvent as agreed to between the purchaser and the supplier. It shall be clear, transparent liquid free from suspended matter, sediments and visible impurities.

4.2 Composition

- 4.2.1 The material shall consist of metallic (cobalt, lead, manganese, zinc, calcium, iron, cerium, barium or zinconium) salts of one of the two organic acids, namely, octoic (2-ethyl hexanoic) acid or naphthenic acid.
- 4.2.2 The octoic acid used in the manufacture of driers shall be of the following specification:
 - a) Acid value 386 to 394.
 - b) Refractive index (at $20^{\circ}C$) 1.424 5 to 1.426 0.
 - c) Moisture content, percent by mass, Max 0.25.
- 4.2.3 The naphthenic acid used in the manufacture of driers shall be of the following specification:
 - a) Acid value 180 to 230.

^{*}Methods of test for ready mixed paints and enamels (second revision).

[†]Glossary of terms relating to paints (revised).

[‡]Specification for petroleum hydrocarbon solvents (first revision).

- b) Unsaponifiable matter, percent by mass, Max 24.0.
- c) Moisture content, percent by mass, Max 0.25.
- 4.3 The material shall also comply with the requirements given in Table 1.

5. PACKING AND MARKING

- 5.1 Packing The material shall be packed in metal containers conforming to IS: 1407-1968*, IS: 2552-1970† or as agreed to between the purchaser and the supplier.
- 5.2 Marking The containers shall be marked with the following:
 - a) Name of the drier, type and grade of the material;
 - b) Manufacturer's name or his recognized trade-mark, if any;
 - c) Mass/volume of the material;
 - d) Batch number or lot number in code or otherwise; and
 - e) Month and year of manufacture.
- 5.2.1 The containers may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5.3 Other details of packing and marking shall be in accordance with the instructions given by the purchaser.

6. SAMPLING

- **6.1** A representative sample of the material shall be drawn as prescribed in 3 of IS: 101-1964[‡].
- 6.2 Criteria for Conformity —A lot shall be declared as conforming to the requirements of this standard if the test results on the composite sample satisfy the requirements prescribed under 4.

^{*}Specification for round paint tins (first revision).

[†]Specification for steel drums (galvanized and ungalvanized) (first revision).

[‡]Methods of test for ready mixed paints and enamels (second revision).

TABLE 1 REQUIREMENTS FOR DRIERS FOR PAINTS

(Clause 4.3)

SL No.	Characteristic Requirement for					METHOD OF TEST, REF TO					
NO.		<i>_</i>	Octoate Driers Naphthenate Driers			5	CL No. IN				
		Solid	Liquid		Solid	Liquid		Appen-			
			Grade 1	Grade 2	Grade 3		Grade 1	Grade 2	Grade 3	dix	No.
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
i)	Colour in 1 cm cell	_		ed to bety er and the				d to between and the		_	9 1 *69
ii)	Viscosity	_	do	do	do	_	do	do	do		17
iii)	Flash point (Abel), Min	_	30°C	30°C	30°C	_	30°C	30°C	30°C		17 6961-261 13 }
iv)	Water content (if water is suspected to be present), percent by mass, Max	1.0	0.5	0.5	0 ·5	1.0	0.5	0.5	0.5	_	14 SI Jo
v)	Metal content, percent by mass:										-
	a) Cobalt	16·0 Min	6·0 ± 0·2	3·0 ± 0·1	-	8·5 Min	6·0± 0·2	3·0 ± 0·1		A -1	
	b) Lead	40.0 Min	24·0± 0·4	18·0 ± 0·3	12.0 ± 0.2	27.0 Min	24·0± 0·4	18·0± 0·3	12·0 ± 0·2	A-2	_
	c) Manganese	16.0 Min	6·0± 0·2	3·0 ± 0·1	_	8.0 Min	<i>i</i> 6·0± 0·2	3·0± 0·1		A-3	
	d) Zinc	18·0 Min	6.0± 0.2	3·0± 0·1	_	10.0 Min	a 6.0 ± 0.2	3·0± 0·1		A-4	
	e) Calcium						3·0± 0·1	2·0 ± 0·1	_	A- 5	_
	f) Iron	12:0 Min	6·0± 0·2	3·0 ± 0·1		8·5 Min	6·0 ± 0·2	3·0± 0·1	_	A- 6	_

7. TESTS

- 7.1 The tests shall be conducted as prescribed in IS: 197-1969* and Appendices A to G. Reference to relevant clauses of Appendices and these standards are given in col 11 and 12 of Table 1 respectively.
- 7.2 Quality of Reagents Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977†) shall be employed in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[Table 1, Item (v)]

DETERMINATION OF METAL CONTENT

A-1. DETERMINATION OF COBALT IN DRIERS

A-1.1 Outline of the Method — The sample is wet oxidized with sulphuric/nitric acid, without any preliminary treatment and cobalt is determined by precipitation of the 1-nitroso-2 naphtholate and subsequent ignition to cobalto-cobaltic oxide (Co₃O₄).

A-1.2 Reagents

- A-1.2.1 Sulphuric Acid concentrated (see IS: 266-1961‡).
- **A-1.2.2** Nitric Acid concentrated (see IS: 264-1976§).
- **A-1.2.3** Hydrogen Peroxide 30 percent solution (see IS: 2080-1962||).
- A-1.2.4 Hydrochloride Acid concentrated (see IS: 265-1976¶).
- A-1.2.5 Sodium Hydroxide Solution 40 percent.
- A-1.2.6 1-Nitroso-2 Naphthol Reagent—to be freshly prepared. Dissolve 4 g of the 1-nitroso-2 naphthol in 100 ml of glacial acetic acid, add 100 ml of water, cool and filter.

^{*}Methods of sampling and test for varnishes and lacquers (first revision).

[†]Specification for water for general laboratory use (second revision).

[‡]Specification for sulphuric acid (revised).

[§]Specification for nitric acid (second revision).

Specification for stabilized hydrogen peroxide.

[¶]Specification for hydrochloric acid (second revision).

- **A-1.2.7** Glacial Acetic Acid See IS: 695-1975*.
- A-1.2.8 Methyl Orange Indicator 0.1 percent solution.

A-1.3 Procedure

- A-1.3.1 Weigh accurately about 5 g of the well mixed sample using a Lunge-Rey pipette or other suitable device into a 500-ml conical flask. Add 25 ml sulphuric acid, mix and heat till charring takes place. Remove from hot plate and add nitric acid cautiously along the sides of the flask (2 ml at a time). Copious brown fumes are evolved. When the reaction slackens, transfer to hot plate and add more nitric acid. Continue heating till no further charring takes place and the solution is pale in colour. Cool, add 50 ml of water and boil gently for a few minutes. Cool and make up to 250 ml in a volumetric flask.
- A-1.3.2 Transfer 50 ml of the solution to a 400 ml beaker. Neutralize using sodium hydroxide solution and methyl orange indicator. Add a few drops of caustic in excess to precipitate the cobalt. Add 10 drops of hydrogen peroxide solution. Warm, add 20 ml of glacial acetic acid, heat gently till the cobaltic hydroxide dissolves. Filter even if no precipitation is apparent into a 600-ml beaker, wash thoroughly with hot water. Dilute to 200 to 250 ml, heat to gentle boiling and add 75 ml of a freshly prepared solution of 1-nitroso-2-naphthol reagent. Stir till the precipitate coagulates and allow to stand for 2 hours at 60°C. Filter through a filter paper (Whatman No. 40 or equivalent) and wash the precipitate with dilute hydrochloric acid (1:19) followed by minimum quantity of hot water to remove hydrochloric acid. Dry the paper and contents in an oven and transfer to a tared porcelain crucible. Ignite carefully in a muffle furnace at 750 to 800°C for 1 hour, cool and weigh as coboltocobaltic oxide (Co₃O₄).

A-1.4 Calculation

Cobalt content, percent by mass
$$= \frac{M_1 \times 73.42}{M}$$

where

 M_1 = mass in g of cobalto-cobaltic oxide, and M = mass in g of sample in the aliquot taken.

A-2. DETERMINATION OF LEAD IN DRIERS

A-2.1 Outline of the Method — The sample is wet oxidized with concentrated sulphuric acid/nitric acid without any preliminary treatment till all organic matter is destroyed. The precipitated lead sulphate is weighed and lead content calculated.

^{*}Specification for acetic acid (second revision).

A-2.2 Reagents

A-2.2.1 Sulphuric Acid — concentrated (see IS: 266-1961*).

A-2.2.2 Nitric Acid — concentrated (see IS: 264-1976†).

A-2.2.3 Dilute Sulphuric Acid — approximately 0.5 N.

A-2.2.4 Ethanol

A-2.3 Procedure — Weigh accurately into a heat resistant beaker, 2 to 2.5 g of well mixed sample using a Lunge-Rey pipette or any other suitable device. Add 20 to 25 ml of concentrated sulphuric acid, mix well and char on a hot-plate. Cover the beaker with a clock glass and add nitric acid through the lip of the beaker cautiously, about 2 ml at a time. Dense brown fumes are evolved. When the reaction has slackened, add more nitric acid, followed by heating till the solution no longer chars and is pale in colour. Cool, rinse down the clock glass and sides of the beaker with water and evaporate to fumes of sulphur trioxide. Cool, repeat the addition of water and evaporation of sulphur trioxide fumes till all traces of remaining nitric acid are expelled. Cool, dilute to 200 ml with water, stir well, and keep aside for at least 12 hours. Filter through a heat resistant sintered glass crucible or a Gooch crucible which has been previously heated at 500°C and tared. Wash the lead sulphate precipitate with 0.5 N sulphuric acid by decantation. Finally transfer the precipitate to the crucible, wash two times more with 0.5 N sulphuric acid and twice with ethanol to remove sulphuric acid. Dry the crucible in an oven at 100 to 105°C for about 1 hour, then ignite at 500°C for about half an hour, cool and weigh the lead sulphate.

A-2.4 Calculation

Lead content (as Pb), percent by mass =
$$\frac{M_1 \times 68.32}{M}$$

where

 M_1 = mass in g of the lead sulphate residue, and M = mass in g of the sample taken for the test.

A-3. DETERMINATION OF MANGANESE IN DRIERS

A-3.1 Outline of the Method — The sample is wet oxidized with sulphuric/nitric acid without any preliminary treatment and the metal estimated in a suitable aliquot by sodium bismuthate method.

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid (sesond revision).

A-3.2 Reagents

- A-3.2.1 Sulphuric Acid concentrated (see IS: 266-1961*).
- **A-3.2.2** Nitric Acid concentrated (see IS: 264-1976†).
- A-3.2.3 Dilute Nitric Acid 0.5 M. Boil concentrated nitric acid in a 250-ml conical flask containing a few boiling chips till absolutely colourless, cool, and dilute 10 to 320 ml with water.
- **A-3.2.4** Standard Potassium Permanganate Solution 0.1 N, freshly prepared.
- **A-3.2.5** Ferrous Ammonium Sulphate Solution Prepare approximately N/10 ferrous ammonium sulphate solution in about 500 ml of water containing 30 ml of concentrated sulphuric acid; cool and make up to a litre with water.

A-3.2.6 Sodium Bismuthate

A-3.3 Procedure

- A-3.3.1 Weigh accurately about 10 g of the well mixed sample using a Lunge-Rey pipette or any other suitable device into a 500-ml heat resistant conical flask. Add 25 ml of sulphuric acid, mix well by swirling and heat gently on a hot-plate till charring sets in; cool slightly, add cautiously nitric acid through the sides of the flask (approximately 2 ml at a time). Dense brown fumes are evolved. When the reaction slackens, transfer the the flask to the hot-plate, heat till the brown fumes have practically disappeared. Continue the additions of nitric acid and heating till the solution becomes pale brown in colour and no further charring takes place on heating. Cool, add 50 ml of water and boil gently for a few minutes. Cool and make up the contents to 250 ml in a volumetric flask.
- A-3.3.2 Transfer 25 ml of the solution to a beaker, add 15 ml of nitric acid and boil for a few minutes. Cool the solution to 15°C, add 1.5 g of sodium bismuthate and stir for 3 minutes or so, maintaining the temperature near 15°C. Filter rapidly, after adding 50 ml of nitrous free dilute nitric acid, into a clean 500 ml Buchner flask using a sintered glass funnel. Keep the solution in the Buchner flask, cool during filtration and subsequent washing. Wash out the beaker and sintered funnel with dilute nitric acid (cool) using about 100 ml in all till the washings are colourless.
- A-3.3.3 Titrate the contents of the flask with ferrous ammonium sulphate solution till the liquid becomes colourless, add 2 ml in excess. Note the total volume added. Immediately back titrate the excess ferrous solution with standard potassium permanganate solution to a faint pink end point. Titrate 25 ml of ferrous ammonium sulphate solution diluted with 150 ml

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid (second revision).

of dilute nitric acid with standard potassium permanganate solution to a faint pink end point. Calculate the strength of the ferrous ammonium sulphate solution.

A-3.4 Calculation

Manganese (as Mn), percent by mass
$$= \frac{(V_1N_1 - V_2N_2) \times 0.109 9}{M}$$

where

 $V_1 = \text{total volume in ml of ferrous solution used,}$

 $\mathcal{N}_1 = \text{normality of ferrous solution used,}$

 V_2 = volume in ml of standard potassium permanganate solution required for back titration,

 \mathcal{N}_2 = normality of standard potassium permanganate solution required for back titration, and

M =mass in g of the sample in the 25-ml aliquot used.

A-4. DETERMINATION OF ZINC IN DRIERS

A-4.1 Outline of the Method — The sample is extracted with hydrochloric acid and the dissolved zinc is titrated with standard potassium ferrocyanide solution using an internal indicator.

A-4.2 Reagents

- **A-4.2.1** Hydrochloric Acid See IS: 265-1976*.
- A-4.2.2 Ammonium Sulphate
- A-4.2.3 Diphenyl Benzidine or Sodium Diphenylamine Sulphonate Indicator
- A-4.2.4 Petroleum Hydrocarbon Solvent 145/205 low aromatic grade (conforming to IS: 1745-1966†).
 - A-4.2.5 Zinc Oxide analytical reagent grade.
 - **A-4.2.6** Sulphuric Acid 3.5 M.
- A-4.2.7 Standard Potassium Ferrocyanide Solution (M/20) Dissolve 21·12 g of potassium ferrocyanide [K₄ Fe (CN)₆ 3 H₂O] and 0·3 g of potassium ferricyanide in water and make up to 1 litre in a standard volumetric flask.

A-4.3 Procedure

A-4.3.1 Weigh 2.034 5 g of dried zinc oxide and dissolve in sulphuric acid. Cool and make up to 250 ml in a standard flask. This solution is

^{*}Specification for hydrochloric acid (second revision).

[†]Specification for petroleum hydrocarbon solvents (first revision).

M/10 with respect to zinc. Pipette 25 ml of this solution into a 250-ml conical flask, and 50 ml of water, 25 ml of sulphuric acid, 3 g of ammonium sulphate and several drops of diphenylbenzidine solution. Titrate the cold solution slowly with vigorous shaking until the colour change from blue-violet to pale green is permanent. Calculate the mass of zinc oxide (ZnO) equivalent to 1 ml of standard potassium ferrocyanide solution.

- A-4.3.2 Weigh accurately about 8 to 10 g of the sample into beaker, dilute with 25 ml of petroleum hydrocarbon solvent, and transfer to a separating funnel using a further 25 to 35 ml of the solvent. Add 40 ml of hydrochloric acid and shake vigorously. Allow to separate and transfer the aqueous layer to a 250-ml graduated flask. Repeat the extraction using half the previous volume of hydrochloric acid, then wash the solvent layer with 30 ml of water, combine the acid extracts and washings in the 250-ml graduated flask, make up to the mark with water and shake well.
- A-4.3.3 Pipette 50 ml of the solution obtained under A-4.3.2 into a 250-ml conical flask. Dilute to about 150 ml with water, add 3 g of ammonium sulphate and 5 drops of diphenylbenzidine or sodium diphenylamine sulphonate indicator. Titrate slowly with standard potassium ferrocyanide solution shaking vigorously until the colour change from violet to pale green is permanent.

A-4.4 Calculation

Zinc (as Zn), percent by mass =
$$\frac{V \times F \times 0.803 \text{ 4} \times 5 \times 100}{M}$$
 where

V = volume in ml of standard potassium ferrocyanide solution required,

F = factor for zinc oxide (ZnO) equivalent to 1 ml of potassium ferrocyanide solution, and

M =mass in g of the sample taken.

A-5. DETERMINATION OF CALCIUM IN DRIERS

A-5.1 Outline of the Method — The sample is wet oxidized with sulphuric/nitric acid without any preliminary treatment; calcium is precipitated as oxalate and titrated with potassium permanganate.

A-5.2 Reagents

A-5.2.1 Sulphuric Acid — concentrated (see IS: 266-1961*).

A-5.2.2 Nitric Acid — concentrated (see IS: 264-1976†).

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid (second revision).

- A-5.2.3 Ammonium Hydroxide Solution relative density 0.9.
- **A-5.2.4** Ammonium Chloride See IS: 1113-1965*.
- A-5.2.5 Ammonium Oxalate
- A-5.2.6 Hydrochloric Acid concentrated (see IS: 265-1976†).
- A-5.2.7 Methyl Red Solution 0.1 percent.
- A-5.2.8 Ammonium Oxalate Wash Solution 0.5 percent.
- A-5.2.9 Standard Potassium Permanganate Solution 0.1 N, freshly standardized.

A-5.3 Procedure — Weigh accurately about 5 g of the well mixed sample using a Lunge-Rey pipette or any other suitable means into a heat resistant 500-ml conical flask. Add 25 ml of sulphuric acid, mix well and char on a hot-plate. Cool slightly, add nitric acid down the sides of the flask (about 2 ml at a time). Copious brown fumes are evolved. When the reaction slackens, heat and continue the addition of nitric acid till no further charring takes place and the solution is pale in colour. Cool, add 100 ml of water and boil gently till all the calcium sulphate has gone into solution. Cool and make up the solution to 250 ml in a volumetric flask. Transfer 100-ml aliquot to a 400-ml beaker, add a few drops of methyl red indicator and neutralize with dilute ammonium hydroxide (1:1) solution cautiously with cooling. Dilute to 250 ml. Add 5 ml of hydrochloric acid, 5 g of ammonium chloride and heat nearly to boiling. Add 2.5 g of ammonium oxalate and heat till the oxalate is dissolved. Add diluted ammonium hydroxide (1:1) with stirring till the indicator turns just yellow. Boil for about 2 minutes and keep aside for 4 hours. Filter through a filter paper (Whatman paper No. 40 or equivalent) (retaining major por tion of the precipitate in the beaker) and wash the precipitate of calcium oxalate several times with dilute ammonium oxalate solution by decantation. Finally transfer the precipitate to the filter paper and wash sparingly with cold water both the beaker as well as the filter paper till the filtrate is oxalate free (test with hot acidified potassium permanganate solution). Place the beaker in which the precipitation was done under the funnel, pierce the filter paper and wash down as much as possible of the precipitate into the beaker with a jet of hot water. Wash the paper with hot dilute sulphuric acid (5 percent) using 200 ml of acid. Finally wash the paper with hot water. Heat the contents of the beaker to 60 to 70°C and titrate with standard potassium permanganate solution to a faint pink end point.

^{*}Specification for ammonium chloride, technical and pure (revised).

[†]Specification for hydrochloric acid (second revision).

A-5.4 Calculation

Calcium (as Ca), percent by mass =
$$\frac{V \times N \times 0.2}{M}$$

where

V = volume in ml of standard potassium permanganate solution required,

 $\mathcal{N} =$ normality of standard potassium permanganate solution, and

M =mass in g of sample in the aliquot taken.

A-6. DETERMINATION OF IRON IN DRIERS

A-6.1 Outline of the Method — The sample is treated to dissolve the iron in hydrochloric acid and the solution is titrated with potassium dichromate in presence of barium diphenylamine sulphonate as indicator.

A-6.2 Reagents

- **A-6.2.1** Stannous Chloride Solution in Hydrochloric Acid Dissolve 30 g of stannous chloride (Sn $\text{Cl}_22\text{H}_2\text{O}$) in 100 ml of hydrochloric acid (relative density 1·18) and dilute to 200 ml with water. Store in a glass bottle over a few pieces of granulated tin.
 - A-6.2.2 Mercuric Chloride Solution 0.2 M.
- **A-6.2.3** Sulphuric-Phosphoric Acid Solution Add 200 ml of concentrated sulphuric acid and 50 ml of syrupy phosphoric acid (88 percent) to about 500 ml of water and dilute to 1 litre.
 - A-6.2.4 Standard Potassium Dichromate Solution 0.1 N.
- A-6.2.5 Barium Diphenylamine Sulphonate Solution 0.25 percent in concentrated sulphuric acid.
- A-6.3 Procedure Weigh accurately about 4 g of the sample into a silica or porcelain basin. Remove the solvent by heating on a hot-plate at low heat at about 110°C. Ignite strongly to remove all carbonaccous matter. Allow the basin to cool and cautiously extract all the iron oxide residue with 5 to 10 ml of hydrochloric acid (relative density 1·18), warming the crucible gently to effect solution. Wash the extracts into a 250-ml conical flask, make the solution up to 250 ml and mix well. Allow any residue in the flask to settle, then pipette 50 ml of solution into a conical flask. Bring to the boil and add stannous chloride solution dropwise until the yellow colour nearly disappears, and add dilute stannous chloride (1 volume of concentrated solution diluted with 3 volumes of water) until the solution is colourless or very pale green. Finally add a few drops in

excess. Cool the solution under the tap, wash down the sides of the flask with water and add 10 ml of mercuric chloride solution. A slight-silky-white precipitate shall appear at this stage. (If the precipitate is dark, start with a fresh aliquot using less stannous chloride.) Dilute to 100 ml. Add 50 ml of sulphuric-phosphoric acid solution and 3 drops of barium diphenylamine sulphonate indicator. Titrate with standard potassium dichromate solution until the green colour first produced deepens to a bluegreen. Continue the titration dropwise from this point. The end-point is distinguished by the formation of an intense blue-violet colouration which remains permanent on shaking.

A-6.4 Calculation

Iron (as Fe), percent by mass =
$$\frac{V \times N \times 0.005584 \times 100}{M}$$

where

V = volume in ml of standard potassium dichromate required,

 \mathcal{N} = normality of standard potassium dichromate solution, and

M = mass in g of sample taken.

A-7. DETERMINATION OF CERIUM IN DRIERS

A-7.1 Outline of the Method — The sample is wet oxidized with sulphuric acid and nitric acid without any preliminary treatment and cerium is determined by first oxidizing Ce (III) to Ce (IV) by ammonium persulphate in presence of Ag+ and then titrating the solution against standard ferrous ammonium sulphate using Fe (II) o-phenanthroline indicator.

A-7.2 Reagents

A-7.2.1 Sulphuric Acid — concentrated (see IS: 266-1961*).

A-7.2.2 Nitric Acid — concentrated (see IS: 264-1976†).

A-7.2.3 Saturated Ammonium Persulphate Solution

A-7.2.4 Fe (II) o-Phenanthroline Solution — 0.025 M.

A-7.2.5 Silver Nitrate Solution — 0.1 M.

A-7.2.6 Standard Ferrous Ammonium Sulphate in N-Sulphuric Acid — 0.1 N.

A-7.3 Procedure

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid (second revision).

- A-7.3.1 Weigh accurately about 5 g of the well mixed sample using Lunge-Rey pipette or any other suitable device into a 500-ml conical flask. Add 25 ml of sulphuric acid, mix and heat till charring takes place. Remove iron hot plate and add nitric acid cautiously along the sides of the flask (2 ml at a time). Copious brown fumes are envolved. When the reaction slackens, transfer to the hot-plate and add more nitric acid. Continue heating till no further charring takes place and the solution is pale in colour. Cool, add 50 ml of water and boil gently for a few minutes. Cool and make up to 250 ml in volumetric flask.
- A-7.3.2 Take 50 ml of the stock solution as prepared in A-7.3.1 in 400 ml beaker. Add 5 ml of silver nitrate solution and 10 ml of persulphate solution. Heat for 10 minutes on a water-bath and then boil the solution on a wire gauze for 15 minutes to destroy the excess of persulphate. Add a drop of the indicator and titrate with standard ferrous ammonium sulphate solution to reddish orange end point.

A-7.4 Calculation

Cerium (as Ce), percent by mass = $V \times 1.401$ where

V = volume in ml of 0.1000 N Fe (II) solution required for titration.

A-8. DETERMINATION OF BARIUM IN DRIERS

A-8.1 Outline of the Method — A known quantity of sample is hydrolyzed in presence of hydrochloric acid and precipitated as barium sulphate. The precipitate is washed, dried and ignited and barium content calculated.

A-8.2 Reagent

- A-8.2.1 Methanol
- A-8.2.2 Dimethyl Sulphate
- A-8.2.3 Hydrochloric Acid concentrated (see IS: 265-1976*).
- A-8.3 Procedure Weigh accurately about 1 g of sample in 250-ml beaker and hydrolyze it completely by heating gently with 100 ml of water and 6 ml of hydrochloric acid for 20 minutes. Cool and filter through a filter paper. Wash with hot water, collecting the washings with the filtrate. Add 20 ml of methanol. Add to the cold solution 0.5 ml of dimethyl sulphate per 10 mg of barium expected to be present (minimum volume to be 2 ml) and immediately place the solution on a steam-bath. The final

^{*}Specification for hydrochloric acid (second revision).

volume of the solution shall be 100 ml. Allow the solution to digest on the steam-bath for 5 minutes. If appreciable decrease in volume occurs, add methanol to restore the 100 ml volume. Filter through a fine ashless filter paper. Wash the precipitate with methanol, with a stream of wash liquid. Dry the precipitate and then ignite in an electric muffle furnace at 750°C for 30 to 60 minutes and weigh as barium sulphate.

A-8.4 Calculation

Barium content (as Ba), percent by mass =
$$\frac{58.85 \ M_1}{M_2}$$

where

 $M_1 = \text{mass in g of barium sulphate obtained, and}$ $M_2 = \text{mass in g of the sample taken.}$

A-9. DETERMINATION OF ZIRCONIUM IN DRIERS

A-9.1 Outline of the Method—The sample is wet oxidized with sulphuric/nitric acid without any preliminary treatment and zirconium determined in a suitable aliquot by precipitation as phosphate in homogeneous solution with metaphosphoric acid and ignition to the pyrophosphate. Under the conditions of the experiment metaphosphoric acid, hydrolyzing, provides the orthophosphate ion to precipitate zirconium as the acid orthophosphate, Zr (HPO₄)². On ignition the pyrophosphate is formed.

A-9.2 Reagents

A-9.2.1 Sulphuric Acid — concentrated (see IS: 266-1961*).

A-9.2.2 Nitric Acid — concentrated (see IS: 264-1976†).

A-9.2.3 Ammonium Nitrate

A-9.2.4 Metaphosphoric Acid

A-9.3 Procedure

A-9.3.1 Weigh accurately about 5 g of the sample using Lunge-Rey pipette or any other suitable device into a 500-ml conical flask. Add 25 ml of sulphuric acid and heat gently till charring occurs. Remove from heat and add nitric acid (about 2 ml each time) carefully along the sides of the flask. Copious brown fumes are evolved. When the reaction has moderated, heat till brown fumes are expelled. Continue addition of

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid (second revision).

nitric acid and heating till no further charring occurs and the solution is pale in colour. Cool, add carefully 5 ml of water along the sides and heat till white fumes appear. Repeat the cooling, addition of water and heating two times more. Cool the contents, add cautiously 50 ml of water and swirl to dissolve any precipitated sulphates. Transfer the clear solution to a 250-ml volumetric flask quantitatively, cool and make up to volume.

A-9.3.2 Transfer a 50-ml aliquot equivalent to approximately 1 g sample to a 250-ml beaker and dilute to 150 to 175 ml with water. Dissolve 5 g of metaphosphoric acid in 25 ml of water in the cold (filtering if any insolubles are noticed) and add to the sample solution. Add 12.5 ml of sulphuric acid, stir and leave overnight. Heat to boiling and keep simmering for 1 hour. Cool to room temperature and filter through a 12.5-cm filter paper (Whatman No. 42 or equivalent). Wash the precipitate with cold 5 percent (m/v) ammonium nitrate solution transferring all the precipitate quantitatively to the filter paper. Wash several times till the runnings fail to show phosphate ion (test with ammonium molybdate). Dry the paper and contents in an oven and transfer to tared silica crucible. Carefully ignite at 900 to 950°C after preliminary charring on hot-plate or burner. Cool and weigh as zirconium pyrophosphate (Zr P_2 O_7).

A-9.4 Calculation

Zirconium (as Zr), percent by mass = $\frac{M_2 \times 34.4}{M_1}$

where

 M_2 = mass in g of the zirconium pyrophosphate, and M_1 = mass in g of the sample in aliquot taken.

APPENDIX B

[Table 1, Item (vi)]

TEST FOR SOLUBILITY IN LINSEED OIL

B-1. OUTLINE OF THE METHOD

B-1.1 A known quantity of the material is mixed with a known quantity of alkali refined linseed oil and examined for solubility.

B-2. REAGENT

B-2.1 Alkali Refined Linseed Oil — conforming to IS: 75-1973*.

^{*} Specification for linseed oil, raw and refined (second revision).

B-3. PROCEDURE

- **B-3.1** Take 5 ml of the sample and 95 ml of alkali refined linseed oil in a 100-ml measuring cylinder and mix well. Allow to stand at room temperature for about 6 hours and examine the condition.
- **B-3.1.1** The requirement of the test shall be taken to have been satisfied if the material dissolves in linseed oil without separating into layers or giving a deposit after 6 hours.

APPENDIX C

[Table 1, Item (vii)]

TEST FOR SOLUBILITY IN PETROLEUM HYDROCARBON SOLVENT

C-1. OUTLINE OF THE METHOD

C-1.1 A known quantity of the material is mixed with a known quantity of petroleum hydrocarbon solvent and examined for solubility.

C-2. REAGENT

C-2.1 Petroleum Hydrocarbon Solvents—145/205 low aromatic (conforming to IS: 1745-1966*).

C-3. PROCEDURE

- **C-3.1** Take 5 ml of sample and 95 ml of petroleum hydrocarbon solvent in a 100-ml measuring cylinder and mix well. Cool to 20°C and examine the condition.
- C-3.1.1 The requirement of the test shall be taken to have been satisfied if the material remains dissolved in the solvent and no precipitation or deposition occurs.

APPENDIX D

[Table 1, Item (viii)]

TEST FOR STABILITY

D-0. GENERAL

D-0.1 The stability of the material at an elevated temperature and when stored open at room temperature has been prescribed.

^{*}Specification for petroleum hydrocarbon solvents (first revision).

D-1. REAGENT

D-1.1 Petroleum Hydrocarbon Solvent — 145/205 low aromatic (conforming to IS: 1745-1966*).

D-2. PROCEDURE

- **D-2.1 Stability at Elevated Temperature** Take one part by mass of the sample with five parts by mass of petroleum hydrocarbon solvent and store in a loosely stoppered glass container in an incubator at $65 \pm 5^{\circ}$ C for 14 days and examine the condition.
- **D-2.1.1** The requirement of the test shall be taken to have been satisfied if the material does not show any observable change like precipitation, deposit or any major change in colour.
- **D-2.2** Stability to Open Storage at Room Temperature Put 100 g of the sample in a 250-ml glass beaker covered loosely with a piece of cloth so as to allow free access to air but exclude dust or other foreign matter from falling into the beaker. Store for 14 days at room temperature and make up the loss of solvent by adding the required quantity of petroleum hydrocarbon solvent and examine the condition.
- **D-2.2.1** The requirement of the test shall be taken to have been satisfied if the material does not show any observable change like precipitation, deposit or increase in viscosity.

APPENDIX E

[Table 1, Item (ix)]

TEST FOR DRYING CHARACTERISTICS

E-1. MATERIALS REQUIRED

- E-1.1 Standard Varnish Prepare a long oil linseed/pentaerythritol/phthalic alkyd varnish using the following ingredients:
 - a) 60 to 65 percent oil length linseed/pentaerythritol/phthalic alkyd solution corresponding to 100 g linseed oil.
 - b) Lead octoate/naphthenate corresponding to 0.5 g lead as metal.
 - c) Gobalt octoate/naphthenate corresponding to 0.05 g cobalt as metal.
 - d) Calcium naphthenate corresponding to 0.1 g calcium as metal.
 - e) Petroleum hydrocarbon solvent 145/205, low aromatic (conforming to IS: 1745-1966*) to reduce the viscosity of the composition to 2 to 3 poise at room temperature.

^{*}Specification for petroleum hydrocarbon solvents (first revision).

- E-1.2 While testing zirconium octoate/naphthenate, lead octoate/naphthenate as given in E-1.1 (b) shall be replaced by zirconium octoate/naphthenate corresponding to 0.5 g of zirconium as metal.
- E-1.3 For preparing standard varnish specified in E-1.1, previously tested and approved samples of driers shall be used. Where such samples are not available samples as agreed to between the purchaser and the supplier shall be used.

E-2. PROCEDURE

- E-2.1 A varnish composition similar to that given in E-1.1 shall also be prepared replacing the approved drier with the drier under test.
- E-2.2 The standard varnish composition (see E-1.1) as well as the varnish composition containing the drier under test shall be kept in a closed container overnight and applied on glass panels by pouring or brushing. The panels after application shall be kept vertically at room temperature and allowed to air dry.
- **E-2.3** The drying time shall be tested as prescribed under 7 of IS: 197-1969*.
- E-2.4 The requirement of the standard shall be taken to have been satisfied if the drying time of the varnish containing the sample under test is not longer than that containing the approved driers.

APPENDIX F

[Table 1, Item(x)]

FREEDOM FROM BITTINESS

F-1. MATERIAL

F-1.1 Glass Panel

F-2. PROCEDURE

- **F-2.1** Take a clean glass panel and spray the sample of drier under test over it. Keep the glass panel in vertical position for a few minutes so as to allow the excess of material to drain off from the panel. Examine the condition of the film of the material so formed.
- F-2.2 The material shall be taken to have satisfied the requirement of the test if there are no specks on the film of the material so formed.

^{*}Methods of sampling and test for varnishes and lacquers (first revision).

APPENDIX G

[Table 1, Item (xii)]

HEXABROMIDE TEST

G-1. REAGENTS

- G-1.1 Chloroform
- G-1.2 Liquid Bromine
- G-1.3 Rectified Spirit conforming to IS: 323-1959*.
- G-1.4 Ether conforming to solvent grade of IS: 336-1973†.
- G-1.5 Hydrochloric Acid See IS: 265-1976‡.
- G-1.6 Alcoholic Caustic Soda Solution approximately 1 N.

G-2. PROCEDURE

- **G-2.1** Take an appropriate quantity of the material and saponify with alcoholic solution of caustic soda. Treat the saponified matter with hydrochloric acid. Decant the fatty matter, wash with hot water to make it free from mineral acid and dry in an oven at 100 ± 5 °C.
- G-2.2 Pipette 1 ml of the fatty matter in a dry test-tube which may be fitted with a ground glass stopper. Add 5 ml of chloroform. Add about 1 ml of bromine dropwise till the mixture is deep red in colour and cool the test-tube in an ice water-bath. Add about 1.5 ml of rectified spirit dropwise while shaking the mixture until the precipitate which is first formed just dissolves and then add 10 ml of ether. Mix and place the tube in the ice water-bath for 30 minutes. Appearance of a precipitate indicates the presence of linseed oil.

Note 1 — The use of Lunge-Rey pipette is suggested for the handling and addition of bromine.

^{*}Specification for rectified spirit (revised).

[†]Specification for ether (second revision).

[‡]Specification for hydrochloric acid (second revision).

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DRYING OILS AND DRIERS

8766-1978 Driers for paints

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1	J	

74-1966	Methods of sampling and test for drying oils for paints (first revision)
75-1973	Linseed oil, raw and refined (second revision)
77-1976	Linseed oil, boiled, for paints (second revision)
79-1975	Linseed stand oil for paints (first revision)
80-1971	Tung oil for paints (first revision)
81-1971	Dehydrated caster oil for paints (first revision)
385-1962	Liquid driers for paints (revised)
386-1962	Liquid driers, concentrated, for paints (revised)